



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 3074

Phthalates in Methanol

This Standard Reference Material (SRM) is a solution of one adipate and six phthalates in methanol intended primarily for use in the calibration of chromatographic instrumentation used for the determination of the certified components in the mixture. Because of its miscibility with water, this SRM can also be used to fortify samples with known amounts of the six phthalates. A unit of SRM 3074 consists of five 2 mL ampoules, each containing 1.2 mL of solution.

Certified Concentration of Constituents: The certified concentration values [1,2] for the six phthalates are given in Table 1. These values are based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using gas chromatography with mass spectrometric detection (GC/MS). A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST.

Supplemental Information: Chemical Abstracts Service (CAS) Registry Numbers of the certified components are listed in Table 1. In addition to the six phthalates listed in Table 1, this solution contains *bis*(2-ethylhexyl)adipate (CAS Registry Number 103-23-1). The concentration of the *bis*(2-ethylhexyl) adipate has decreased since the preparation of this solution, so it is not included as a certified concentration.

Expiration of Certification: The certification of this SRM lot is valid until **31 July 2013**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). However, the certification is nullified if the SRM is damaged, contaminated, or modified. NIST reserves the right to withdraw, amend, or extend this certification at anytime.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive surface changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Coordination of the technical measurements leading to the certification was under the direction of M.M. Schantz and S.A. Wise of the NIST Analytical Chemistry Division.

Partial support for the preparation and certification of this Standard Reference Material was provided by the U.S. Environmental Protection Agency Office of Water, Office of Enforcement and Compliance Assurance, and Office of Research and Development.

Analytical measurements of the SRM were performed by B.A. Benner, Jr., C.R. Mack, and L.K. Walton of the NIST Analytical Chemistry Division.

Preparation of the SRM was performed by B.A. Benner, Jr. and M.P. Cronise of the NIST Standard Reference Materials Program.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh of the NIST Statistical Engineering Division.

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Certificate Issue Date: 11 May 2006
See Certificate Revision History on Last Page

The support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

INSTRUCTIONS FOR USE

Handling: This material contains phthalates and an adipate and should be handled with care. Use proper disposal methods.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures lower than 30 °C.

Opening of Ampoule: Open ampoules carefully to prevent contamination and injury. The ampoules are pre-scored and should **NOT** be opened using a file. Sample aliquots for analysis should be withdrawn at 20 °C to 25 °C **immediately** after opening the ampoules and should be processed without delay for the certified value to be valid within the stated uncertainty. Because of the volatility of methanol, certified values are not applicable to material stored in ampoules that have been opened for more than 5 minutes, even if they are resealed.

PREPARATION AND ANALYSIS

SRM Preparation: The six phthalates and one adipate used in the preparation of this SRM were obtained from a commercial source. The solution was prepared at NIST by weighing and mixing the individual components into the methanol. The weighed components were added to the methanol and mixed until completely dissolved and homogenized. The total mass of this solution was measured and the concentrations calculated from this gravimetric procedure. These gravimetric concentrations were adjusted for the consensus purity estimation of each component, which were determined by using capillary gas chromatography with flame ionization detection and differential scanning calorimetry. This bulk solution was then chilled to approximately –5 °C and 1.2 mL aliquots were dispensed into 2 mL amber glass ampoules, which were then flame sealed.

SRM Analysis: Aliquots from nine ampoules, selected according to a stratified, random sampling scheme, were analyzed in duplicate by using GC/MS employing an immobilized nonpolar (5 % phenylmethylpolysiloxane) stationary phase column (see Figure 1). An internal standard solution containing six ring-*d*₄ phthalates was added to each sample for quantification purposes. Calibration solutions consisting of weighed amounts of the six phthalates and one adipate and the ring-*d*₄ phthalate internal standard compounds in methanol were chromatographically analyzed to determine analyte response factors.

Table 1. Certified Concentrations of the Six Phthalates in SRM 3074

Compound	CAS Registry Number ^(a)	Concentration	
		mg/kg ^(b)	mg/L ^(c)
Dimethylphthalate	131-11-3	55.6 ± 1.2	44.0 ± 0.9
Diethylphthalate	84-66-2	51.4 ± 1.7	40.7 ± 1.4
Di- <i>n</i> -butylphthalate	84-74-2	51.2 ± 1.2	40.5 ± 0.9
Benzylbutylphthalate	85-68-7	52.2 ± 1.4	41.3 ± 1.1
<i>Bis</i> (2-ethylhexyl)phthalate	117-81-7	58.6 ± 1.3	46.4 ± 1.0
Di- <i>n</i> -octylphthalate	117-84-0	48.2 ± 1.4	38.2 ± 1.1

^(a) Chemical Abstracts, Thirteenth Collective Index, Index Guide, American Chemical Society, Columbus, Ohio, 1996.

^(b) The results are expressed as the certified value ± an expanded uncertainty. The certified value is the unweighted average of the concentrations determined by gravimetric and chromatographic measurements. The expanded uncertainty, at the 95 % level of confidence, is calculated as $U = k u_c$ where u_c is a combined standard uncertainty calculated according to the ISO and NIST Guides [3] and $k = 2$ is the coverage factor. The value of u_c includes both a correction for estimated purity and an allowance for differences between the concentrations determined by gravimetric preparation and chromatographic measurements.

^(c) The concentration in mg/L was obtained by multiplying the certified value, expressed as a mass fraction, by the measured density of the SRM solution at 22 °C (0.7914 g/mL). This concentration is for use over the temperature range of 20 °C to 25 °C, and an allowance for the change in density over this temperature range is included in the uncertainty.

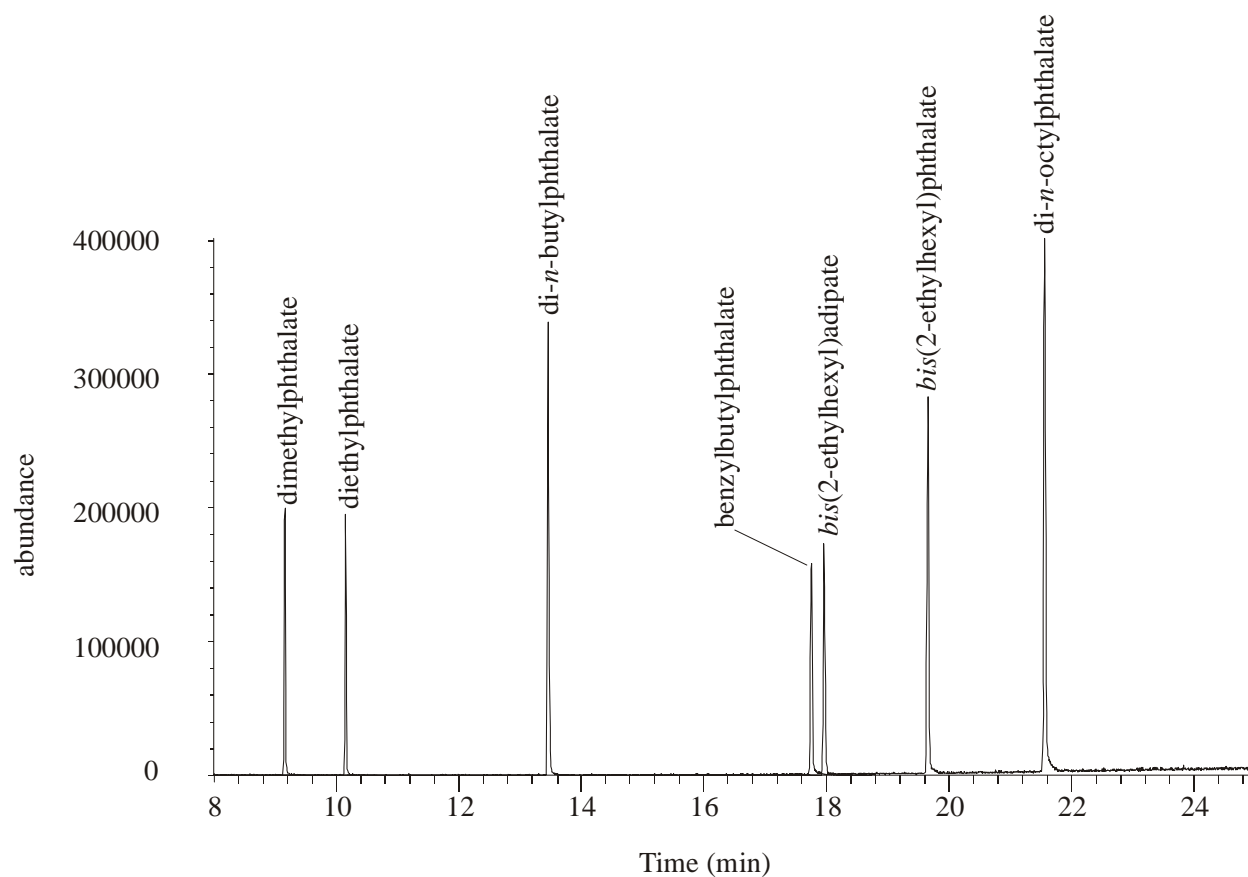


Figure 1. Chromatogram from GC/MS analysis of NIST SRM 3074 using a 0.25 mm i.d. \times 60 m (length) fused-silica capillary column with a 5% phenylmethylpolysiloxane phase (0.25 μ m film thickness). Temperature Program: 100 $^{\circ}$ C (1 min) then 30 $^{\circ}$ C/min to 200 $^{\circ}$ C, then 7.5 $^{\circ}$ C/min to 320 $^{\circ}$ C; MS scan from 50 m/z to 300 m/z (2.4 scans/s), electron multiplier voltage = 2000 V.

REFERENCES

- [1] May, W; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136, U.S. Government Printing Office: Washington, DC (2000).
- [2] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (1995).
- [3] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at <http://physics.nist.gov/Pubs>.

Certificate Revision History: 11 May 2006 (Editorial change); 19 April 2006 (Removal of the adipate component from the list of certified constituents and extension of certification period); 20 February 2003 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.